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Laboratory Director Approval:

| Laboratory Director Approval: | Lock Teller | 08/26/2021 |
| Jeffney Moone 08/26/2021 |
| QA Manager Approval: | Lock Teller | 08/26/2021 |

Laboratory Internal Audit Standard Operating Procedures

Access to this SOP shall be available within the laboratory for reference purposes; the official copy of this SOP resides on the official Georgia EPD website at

https://epd.georgia.gov/about-us/epd-laboratory-operations. Printed copies of this SOP will contain a watermark indicating the copy is an uncontrolled copy.

1. Scope and Application

- The Quality Assurance department conducts an internal audit of the entire group of laboratories on an annual basis. This is both a system and a method audit combined. It is conducted by one or more personnel with experience in auditing techniques over a period of several weeks. Each laboratory will have an individualized audit with checklists for a general walkthrough and all methods pertaining to that laboratory. Each method is audited and discussed in detail with the responsible managers, supervisors and analysts. All deficiencies require a response outlining correcting the problem with 30 days of their receipt. A follow up audit will be performed to confirm that any changes, indicated in these responses, are complete.
- Audit results are kept on file for review by inspecting authorities, primarily 1.2 USEPA, when performing an external audit on the GAEPD Laboratory.

2. Definitions

Refer to Chapter 3 of the GAEPD Laboratory Quality Assurance Plan (LQAP) for 2.1 Quality Control Definition.

3. Quality Control

- Each procedure that has a definitive method will have a checklist listing major aspects of the analysis that require scrutiny.
 - All checklists are based on appropriate methodology, USEPA protocols, NELAP or requirements stated in the GAEPD LQAP. If any interpretation of a method differs from the checklist, it is discussed with the QA Manager. A valid argument will be indicated in the comments section of the checklist and if necessary, a change made to the checklist for future audits. However, all checklists are considered "locked" for the current audit. This removes any confusion during the period of the audit.
- 3.2 Checklists are not "controlled documents," but have limited access, in a read-only format.

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| 4 | Pro | ce | dі | ıre |

- 4.1 A facility wide internal audit of all laboratories is required by USEPA (per NELAP) on an annual basis. This is an all-encompassing audit including methodologies and systems. The Quality Assurance (QA) Manager is responsible for all parts of the process. 4.2 Laboratories are given at least two weeks' notice by email and staff meetings to inform the Laboratory Manager of specific analyses targeted for quality system compliance. Submitted samples will be relatively current and reviewed under the most recent method performance criteria. 4.3 When a method is used for both Drinking Water and Water Quality purposes, the GAEPD lab will default to Drinking Water samples for method review. 4.4 The QA Manager will gather all audit documentation filed in the QA office. All controlled documents for the method will be reviewed and compared to the original Quality Systems documentation by the QA Manager. 4.5 All requested sample data will be checked for the following: Quality control limits in Quality System documentation compared with those 4.5.1 currently established in LIMS. 4.5.2 Calibration information, including instrument calibration and calibration log, if different from sample analysis run and the standard preparation log for calibration standards. Sample preparation and/or extraction logs. Sample run logs, which includes any instrument run logs that reference 4.5.4 that sample ID. 4.5.5 Raw data, including all chromatograms, reports, calculations, spreadsheets, etc. 4.5.6 Instrument maintenance log for the period of sample analysis. 4.5.7 Any additional information necessary to validate sample analysis as required by the QA Manager. 4.6 The method auditor will use a QA generated checklist to review the documentation provided. Each laboratory will randomly choose and submit a recent data set for each method for auditing. Any entry in the findings will be classified as one of the following: 4.6.1 Deficiency: A procedure is not followed in accordance with established protocols. This is considered a serious problem and must be corrected
- completion.

 4.6.2 Recommendation: A procedure is either awkward, incomplete or non-existent. The recommendation is made to help the situation, if implemented. This is a suggestion, not a requirement of the method or procedure in question, simply more information or clarification that will aid the analyst

immediately. A written response to the QA Manager is required from the responsible Laboratory Manager, indicating the corrective action and date of

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- and/or data reviewer. If the Laboratory Manager chooses not to implement the recommendation, a response is helpful, but not required.
- 4.6.3 Clarification: A response is required to clarify a confusing situation. An example would be checking for an additional surrogate, including calibration and not including it in the final report.
- 4.6.4 Comment: A notation about an issue. A comment is usually a request for more information to explain a situation found during the audit.
- 4.7 Each Method Checklist (see Appendix A) will have extensive and exclusive questions about each method or group of methods used in the laboratory. The general walkthrough checklist (see Appendix B) will cover systems checks such as balances, refrigerators, properly labeled bottles, etc. If the audit reveals a negative answer to a method requirement, the auditor will write a D, R or C for Deficiency, Recommendation or Clarification, respectively along with any appropriate comments for that question. A reference to the section in the method in question should be made if possible.
- If an EPA method checklist is not available for a particular method, the auditor will use the method and associated SOP to ensure that all QA/QC requirements are being met. It will be noted on the general walkthrough checklist that an EPA method checklist did not exist or could not be found. There are two techniques used to conduct each internal audit. One is to interview each analyst and/or supervisor, asking them questions from the checklist. Inability to answer questions about the procedure is an indication of possible problems. Consideration must be given to the time an analyst/supervisor has performed the procedure. However, if they have more than a month of actual data producing experience, they should be knowledgeable in the procedure's finer points. It is perfectly acceptable for them to refer to their SOP for guidance. It is not appropriate to have a

4.9 There are two categories of checklists as mentioned above, Method Checklist (See Appendix A) and General Walkthrough (See Appendix B). In addition to the guidance of the checklist, use the following for each type:

aspects of the procedures.

generated "cheat sheet" for reference. The second technique is a more limited contact with the analyst and/or supervisor and focuses on data review. This requires a larger number of sample data pulled and checked thoroughly against method checklists and methods themselves. A good routine is to alternate between the two techniques each year to cover all

- 4.9.6 Method: Method checklists take up most of the time. It involves most of the facets covered in each method or QAP procedure. If the analysts and/or supervisors are to be interviewed, see Section 4.8, the following is the typical routine:
- 4.9.6.1 Check training records for current Initial Demonstration Forms (IDFs) or Continuing Demonstration Forms (CDFs).

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| 4.9.6.2 | Check the sample data provided for that method. This includes all preparation logs, run logs and QC associated with the data. |
|-----------|--|
| 4.9.6.3 | Check maintenance logs for any instrumentation associated with the |
| | method. |
| 4.9.6.4 | Determine traceability of several associated reagents and standards. |
| 4.9.6.5 | Check run logs for the following: |
| 4.9.6.5.1 | Up to date |
| 4.9.6.5.2 | Pages numbered and consecutive |
| 4.9.6.5.3 | List all analyses analyzed during the day. Not an abbreviated list of samples only. |
| 4.9.6.5.4 | Pages initialed or identified in the header with the initials and date. Hand initialing is preferred. |
| 4.9.6.5.5 | Each run has the appropriate identification for standards, blanks, spikes and samples. |
| 4.9.6.5.6 | The proper number of standards are analyzed, bracketing samples per method requirements. |
| 4.9.6.5.7 | A curve is analyzed with the proper number of standards analyzed. If an abbreviated number are analyzed, investigate. |
| 4.9.6.5.8 | Proof of a second source to double check the curve standards' concentration. |
| 4.9.6.5.9 | Each standard has a unique identifier and is traceable from the logbook. Just because the header has a lot number does not necessarily mean the standard is traceable from this point. Make sure there is not a break in the chain of traceability. Note: Pay particular attention to standard lot numbers that are "hard copied" on all forms. They are notorious for being neglected when a new standard is prepared. This practice should be avoided and discouraged. |
| 4.9.6.6 | Pick at least one standard from the run log (typically the QC spike standard). More is preferrable. Trace them to the containers that hold them. Trace that to the standard prep log. Then check for the following in the standard prep log: |
| 4.9.6.6.1 | All fields are filled in with the appropriate entry or N/A. |
| 4.9.6.6.2 | Write-overs and scratch-throughs. |
| 4.9.6.6.3 | Preparation charts for multiple component standards. |
| 4.9.6.6.4 | Certificates of Analysis. There must be an appropriate "CoA" for each standard. Verify that the standard matches the information in the logbook and notice how the CoAs are organized. They should be easily accessible for review and properly filed, preferably in a 3-ring binder. |
| 4.9.6.6.5 | Each container involved with the current standard is properly marked for easy identification. It is NOT acceptable to mark a tray with the standards in it. There must be the name of the standard and the lot number of all containers involved. Use the example, "If the containers are dropped and scrambled, can the standard still be identified?" |

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| 4.9.6.7 | Take the laboratory SOP and compare it to the appropriate method approved by the EPA. All QC, initial calibration, sampling, data calculations and standard information must be covered in the SOP. Also, detailed instructions for LIMS entry or reference to another SOP with LIMS instructions should be included. |
|---------|--|
| 4.9.6.8 | Each method will have certain specifics, especially with QC parameters, that must be covered. These may include such items as specific surrogates, initial calibration concentration order, concentrations for continuing calibration verifications (CCVs), number of samples between CCVs, recovery acceptance criteria, etc. If there are exceptions to the method, they must be clearly documented with adequate reasons provided for the change with a corrective action document. Check previous audits to see if this issue has been addressed before. A previous deficiency must be corrected. If not, it is a serious violation and will generate multiple deficiencies. |
| 4.9.7 | General Walkthrough: At the beginning of each laboratory audit, a walkthrough of the laboratory area is made using the checklist of the same name. General items such as reagent bottles, carboys, balances, |
| CC | refrigerators, etc. are checked and noted if found out of compliance for any type of infraction. Although safety issues are not specifically part of the audit, it is important to make sure obvious safety violations are also noted and corrected. |
| 4.10 | <u>Data Review</u> |
| 4.10.1 | Two weeks or more before an audit of a laboratory, the QA Officer must send a notice of intent to audit. With this notice, a list of requested analytical data sets are emailed to the laboratory manager. All data pertaining to these data packages must be available for review at the time of the audit. They should include at a minimum: |
| 4.10.2 | Raw printouts (reports or chromatograms) |
| 4.10.3 | Calibrations |
| 4.10.4 | Logbook copies |
| 4.10.5 | Extraction or Digestion logbook copies if applicable |
| 4.10.6 | Spreadsheets or other data sheets used for calculations |
| 4.10.7 | Any additional information, such a CoCs, etc. if readily available |
| 4.10.8 | Any relevant checklists |
| 4.10.9 | Most laboratories have the data in a file folder with the appropriate sample IDs and/or batch numbers labeled on the tab. Ease of review is essential. If a sample package is disorganized or missing crucial information, it will be difficult if not impossible to review. Comments, Recommendations and/or Deficiencies should be issued for failure to provide adequate information to the QA Officer. |
| 4.10.10 | Check each section in reference to the method/SOP which should be the |

same. If there are any discrepancies, they should be noted as

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| | deficiencies. It is best to start with the Initial Calibration and continue from |
|--------|---|
| | there. Check a minimum of the following: |
| 4.11 | Initial Calibration |
| 4.11.1 | Reporting Limit(s) must be at or above the lowest point on the calibration curve. |
| 4.11.2 | All compounds have a valid curve. There are exceptions but only method specific exceptions are allowed. |
| 4.11.3 | The minimum number of points must be analyzed. If a point is missing, a written reason why must be included. This will typically be a Deficiency regardless of the reason. |
| 4.11.4 | The date of the Initial Calibration must be earlier than any analyses associated with it. |
| 4.11.5 | The value indicating a "valid" curve, whether Correlation Coefficient, r ² or %RSD must be calculated and available as part of the curve folder. An auditor should never be expected to calculate any of these values. |
| 4.11.6 | A plot of each compound's "curve" should be available. Usually for large numbers of compounds, i.e., 8270, these are not plotted but are available for review at any time. |
| 4.11.7 | A second source standard should also be analyzed before the curve is approved. It should contain all compounds of interest. If any compound is missing due to an odd mixture of compounds in a standard or some other reason, the missing compounds may be analyzed via a second ICV standard. If certain compounds are missing from an ICV and are not accounted for in a separate ICV standard, a corrective action is required to explain the missing ICV compounds. Missing ICV compounds, unless allowed by the method, will generally be a Deficiency. |
| 4.11.8 | The values of the second source should be the same as that of a continuing calibration verification unless specified otherwise by the method. |
| 4.12 | Continuing Calibration Verification |
| 4.12.1 | Continuing Calibration Verifications (CCVs) are standards that are analyzed at the beginning of an analysis run or batch to verify the current initial calibration. Each method details the number of samples or QC standards analyzed between CCVs, but the beginning of the analysis run or batch always has a CCV analyzed before samples, blanks, etc. The only exception is if a calibration curve was analyzed within the same batch of samples and is the beginning of the analysis run. In this case, the appropriate standard concentration is recalculated against the curve, |
| | generating numbers that should pass the CCV requirements set by each |

method. If it does not, the initial calibration is not valid for analysis and

another curve must be generated.

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- 4.12.2 Most methods require an ending CCV to bracket all samples within a set of CCVs. However, some methods, notably 8260 and 8270, do not require an ending standard. Make sure the method requirements are followed.
 4.12.3 Most methods allow for individual compounds to be higher than the allowed %D if there are no reportable results for that compound during the analysis run. However, this is a rule mainly for troublesome compounds. Occasionally a compound will not pass. The waiver applies in this situation. If most compounds are running high, this indicates the curve is failing and the analysis should be repeated with a new calibration, if possible. As stated in SW-846, this rule is for the occasional exception, not as an excuse to provide bad data.
 4.13 Laboratory Control Spikes
- 4.13.1 Every preparation batch of samples should have a Laboratory Control Spike (LCS) and a Laboratory Control Spike Duplicate (LCSD) unless specified otherwise by the method. These spike standards are prepared in an inert media that represents the matrix of the samples in the batch (DI water for liquid samples, a non-reactive solid for solids/wastes, Ottowa sand for example). The process of sample preparation must be the same for the LCS/LCSD. This proves that the procedure is in control. If the parameters are not met for acceptable results of LCS or LCSD, the procedure is out of control. All sample results associated with the LCS and LCSD are considered estimated.
- 4.13.2 There is usually an exception. Most methods allow for the LCS %recovery to be higher than the upper limit of acceptability if there are no positive results for any associated sample for high bias compounds. Note: The GA EPD Lab generally requires all LCS %recoveries to meet method LCS criteria except for troublesome compounds associated with hazardous waste analyses.
- 4.13.3 LCSD %recoveries are not considered a part of QC criteria. The sole purpose for the LCSD result is calculating precision. If any compound exceeds the Relative Percent Difference (RPD) acceptable limit, the procedure is considered out of control and all results, including not detected, are flagged as estimated (with a "J") and commented in Labworks.
- 4.13.4 Some analyses allow for the CCV to also be the LCS. The compounds listed for the LCS are the primary concern, in this case. If a non-LCS compound is outside acceptable limits for the CCV and is high, only positive results for that individual compound are flagged as estimated ("J"). If a non-LCS compound is lower than the acceptable limit, this usually indicates a failing curve. But there are "trouble" compounds that are exceedingly difficult to keep stable. In SW-846 for example, the compounds that are gases at room temperature are very unstable. EPA allows for these compounds to fail, flagging any positive result with a "J".

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Otherwise, the expense of constant recalibration of the entire compound list is prohibitive.

4.14 Matrix Spikes

Every preparation batch of samples must have a Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) analyzed unless otherwise specified by the method. A MS and MSD are aliquots of a designated sample, spiked with a standard. The recovery of this standard determines the accuracy (MS) or the precision (MSD). These spikes are prepared exactly as the samples in the batch are prepared. The information gained from these QC samples determines if there are contributing interferences from the sample matrix that will cause an erroneous reading. If a MS or MSD "fails", falls outside acceptable QC limits, it does not invalidate the data. However, careful interpretation of the failures and any other indications, such as surrogate failure, must be made to determine if an influence of data from the matrix is severe enough to note. While this is traditionally a "customer" decision, usually the customer relies on the lab's expertise to guide them on data reliability.

4.14.2 M

MS/MSD preparation should be at the same time as the LCS/LCSD, Blank and samples for the batch. The results for the MS/MSD determine the % recovery for all spiked compounds. This may be all the compounds in the analyte list or a method specific mix of compounds that represent a large constituent of analytes analyzed for by the method.

- 4.14.3 If there are high levels of target analytes, especially spike analytes, the MS/MSD may fail due to interferences from these analytes or be diluted out. This does not invalidate the data. It does require a corrective action comment and a flag for the final report.
- 4.14.4 If there are sufficient interferences in the sample other than target analytes, the MS recovery or MSD precision may also fail. The data is still acceptable but a flag stating that the MS and/or MSD failed for the appropriate compounds due to high levels of non-target compounds must be on the final report. A corrective action is also needed. Similarly, if the interferences are significant, the sample will have to be diluted. If the dilution is high enough, the actual concentration of the Matrix Spike compounds will fall below their Reporting Limit. If this is the case, a comment is made in Labworks that notes the MS/MSD was diluted to the point that spike concentrations fell below the Reporting Limit. Note that this does not constitute a corrective action. Only a comment under the QC sample Test Code for that analysis.
- 4.14.5 Many times there is not enough sample volume or mass to prepare a Matrix Spike and Matrix Spike Duplicate. When this happens, it generates a corrective action. This corrective action will be referenced in a Labworks comment under the QC Sample Test Code for that analysis (the \$S_ code not the \$R_ code) and these will have to be removed from Labworks to

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close out the batch as completed. The comment will simply state that there was insufficient sample volume or amount for a MS/MSD. 4.15 Method Blanks 4.15.1 Each batch of samples must have a Method Blank prepared along with the samples and associated QC (LCS/LCSD, MS/MSD). A Method Blank is an uncontaminated medium like the medium of the samples (DI water for liquids, a non-reactive solid for solids/wastes, Ottowa sand for example) prepared exactly as the samples are prepared with all reagents. surrogates, internal standards, etc. It must complete the process in the same time frame as the samples in the batch and, when analyzed, have no target compounds above their Reporting Limits. 4.15.2 If a Method Blank is contaminated, there are several options. 4.15.3 If there is sufficient sample amount and holding time, the samples should be prepared again. This is rarely an option. 4.15.4 All compounds above the Reporting Limit are flagged with a "B". This indicates that any positive results are questionable, due to possible contamination from the laboratory. If there are no positive compounds in the samples, no further action is 4.15.5 required other than a Corrective Action. Obviously, the Blank contamination is the only contamination in the sample batch. If the compound's concentration found in a sample is ten times greater or more than the blank contamination concentration for that compound, it is considered insignificant and the result is not flagged. 4.16 Internal Standards 4.16.1 Many organic analyses have internal standards. These special compounds meter how effectively the target compounds are removed from the sample. They must fall within a specific range to extraction or digestion bias. 4.16.2 Internal Standard % response values should be calculated (either by hand or instrument software) on the chromatogram, data spread sheet or another accompanying sheet. An auditor should never have to calculate them. It indicates the analyst did not bother to check them on a day-to-day basis as is required. 4.17 Raw Data 4.17.1 Raw data will vary widely depending on the type. Organic and GCMS results have a tremendous number of printouts per sample, while inorganics will only have a couple of logbook sheets. This makes the use of checklists very important. Each laboratory's checklist is specific to the general requirements of all similar methodology. For example, there are Drinking Water and Hazardous Wastes checklists for Organics. The former is designed to cover the QC required for Drinking Water samples and the

latter for Hazardous Waste (SW-846) methods. Follow these checklists closely. It is highly recommended that the accompanying method be reviewed and important points be highlighted to assist in covering

4.17.2

4.17.3

4.17.4

4.17.6

4.17.7

4.17.8

4.18 4.18.1

4.18.2

4.18.3

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| exceptions that each method will possess. Also, notes in the margins of the method and at the end of the checklist are additional ways to refine the checklist for each requirement. If there were previous deficiencies, it is also convenient to reference them on the checklist to determine if the problem persists. However, there are certain general items, listed below, related to data that should be scrutinized: |
|--|
| Any notations made must be initialed and dated. If the printout has the analyst's initials printed, it may or may not suffice. Usually, it does not, because there is no way to prove if the analyst made the notation. All logbook pages must have proof of pagination. They must have the initials of the analyst somewhere on the sheet also. If there are entries for more than one analyst, each must be identified. Note: Labworks will only allow one set of initials on a final report. Protocol should be the senior analyst or supervisor typically. |
| Make sure that any LCS, MS, calibration curve, continuing calibration verification and/or independent calibration standards have the lot number easily traceable. |
| Any calculations must accompany the data in some form. It can be from a spreadsheet, calculated on the raw data page or calculated by the instrument software. However, it must be initialed and dated somewhere |
| on the page. It is imperative that the identity of the analyst, supervisor or manager be known for future scrutiny. All associated logbook pages must have all blanks filled in. If there is no data for a box or blank, an "N/A" or dash (whichever is most appropriate) should be in the space. |
| All reagents used must have lot numbers that are traceable to either a reagent logbook or directly to a Certificate of Analysis for that reagent. If there is an expiration date listed, check to make sure the date has not been exceeded. This is an oversight for most laboratories. |
| Generally, there should be a maximum of 10 injections between CCVs for automated instrumentation. Especially for Drinking Water samples. There are exceptions such as 8000 methods for GC and GCMS with run on the 12-hour clock. In this case, clock starts with the first definitive standard, the initial CCC for GC and the Tune for GCMS. The last acceptable injection |
| must be less than 12 hours from this time. <u>Audit Conclusion</u> When the internal audit is finished for the laboratory, a final report is generated. It will be broken down into the following sections (See |
| Appendix C for the final report form): Section I: Instructions and Definitions: Gives and introduction and defines |

each of the possibilities that the findings will list.

Section II: General Walkthrough: Any findings from this part of the audit are placed here. They are not method specific in most cases but are EPD

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| | LQAP, NELAP or "Best Practice" requirements. They usually cover system requirements versus method requirements. |
|--------|--|
| 4.18.4 | Section III: Methods: Each analytical method covered by the audit for each laboratory/department is listed here with findings or a statement "no deficiencies or recommendations." Any finding should be specific to that method. |
| 4.18.5 | Section IV: Laboratory-Wide Deficiencies and Recommendations: Any general findings that apply to several aspects of a laboratory but are not covered by the walkthrough, such as incorrect logbook entries for several methods or lack of initials for several different analytical logbooks, are listed here. |
| 4.18.6 | Once the final report has been submitted to the managers and supervisors of the various laboratories/departments, an Internal Audit Deficiency Response Form (see Appendix D) must be completed for each deficiency or recommendation along with a corrective action. (Either a corrective action form or an outline of corrective actions to be taken, whichever is most appropriate.) |
| 4.18.7 | A timeframe will be issued by the QA Officer for when the corrective actions must be completed. (The response form must be completed within 30 days.) This is dependent on the complexity of the corrective action needed. Simple corrections will be required to be implemented immediately. Deficiencies requiring more involved processes will be given a longer time limit to correct the problem. |
| 4.18.8 | A mini follow-up audit will be performed to verify that corrective action procedures have been put into place to correct any deficiencies. |

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Appendix A: Method Checklist

Drinking Water Checklist

Method 245.1 Rev 3.0, 1994

Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry page 1 of 3

The following checklist is a <u>guide</u> for Mercury Analysis with EPA Method 245.1 rev.3.0. If a discrepancy is found between the Method and the checklist, the Method is considered correct.

| Auditor(s): | *. | | |
|--|-----------|----------|----------|
| - | | | |
| Query | Reference | Y, N, NA | Comments |
| 1. Is the dead air space in the digestion vessel (BOD bottle) purged before the addition of stannous chloride solution, to remove volatile interferences? | Sec 4.2 | | |
| 2. Does the Laboratory have a suitable Cold Vapor AA system for analysis? This includes: a. AA spectrometer | Sec 6.1 | ПЕ | ed C |
| b. Mercury Hollow Cathode Lamp c. Absorption Cell d. Air pump with tubing e. Data integrator (chart recorder, computer, etc.) | 2 26 40 | | |
| f. Water bath at 95°C with depth of 2 - 3 inches for sample solution coverage during analysis. g. Analytical balance to 0.0001 g if used for preparing standards and reagents. | | | |
| If self-contained analyzer is used, make sure it satisfies the necessary requirements of the method. | | | |
| 3. Does the Laboratory have a water bath with a covered top capable of maintaining a water depth of 2-3 inches at 95°C? | Sec 6.3 | | |
| 4. Are the Reagents used for sample preparation mercury free (in particular, the acids)? | Sec 7.1 | | |



Laboratory Name:

Location:_____
Date of Audit:__

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Drinking Water Checklist

Method 245.1 Rev 3.0, 1994

Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry page 2 of 3

| 5. Are there three types of blanks, calibration, laboratory reagent (LRB), and laboratory fortified (LFB) blanks used during the analysis process? | Sec 7.11 | | | |
|---|-----------|----|----|----------|
| 6. Are samples to be analyzed for total mercury acidified on site or immediately upon receipt with 1+1 Nitric Acid to a pH < 2? | Sec 8.2 | | | |
| 7. Are samples held for 16 hours and checked again for a pH of < 2 before analysis or storage? | Sec 8.2 | | | |
| 8. Are samples analyzed within 28 days of collection? | Sec.8.2 | | | |
| 9. Has an Initial Demonstration of Performance been performed (Linear Dynamic Range (LDR), Quality Control Sample (QCS), and MDL study), by each analyst? | Sec 9.2 | ПС | 5U | |
| 10. Is a QCS analyzed after each calibration curve or at least once a quarter and does it fall within ± 10% of the true value? | Sec 9.2.3 | | | 0.00.000 |
| 11. Has a Laboratory Reagent Blank (LRB) been analyzed with each batch of 20 or less samples and does each pass? | Sec 9.3.1 | | | |
| 12. Has a Laboratory Fortified Blank (LFB) been analyzed with each batch of samples and does it pass (85-115%)? | Sec 9.3.2 | | | |
| 13. Does the Laboratory have control charts based on the recovery of the LFB and are they within the 85-115% control limits? | Sec 9.3.3 | | | |
| 14. Does the Laboratory analyze a | Sec 9.4.1 | | | |



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Drinking Water Checklist

Method 245.1 Rev 3.0, 1994

Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry page 3 of 3

| Laboratory Fortified Matrix (LFM) after 10% of samples or 1 per sample set of 10, and does it pass (70-130%)? | | | V | |
|--|---------------------|----|----------------|---|
| 15. Is the instrument set to 253.65 nm? | Sec 10.1 | | | |
| 16. Does the Laboratory use a 100 ml aliquot of sample and add: 5 ml of H_2SO_4 2.5 ml of HNO_3 15 ml of $KMnO_4$ or more 8 ml of $K_2S_2O_8$ to each sample? | Sec 11.1.1 - 11.1.3 | | | |
| 17. Does the Laboratory heat the samples in a water bath to 95°C for 2 hours? | Sec 11.1.3 | IE | 9 0 | C |
| 18. Does the Laboratory add 6 ml of NaCl-(NH ₂ OH) ₂ • H ₂ SO ₄ to reduce excess permanganate? | Sec 11.1.5 | λ. | | |
| 19. Does the Laboratory use 5 concentrations other than zero for its curve? | Sec 11.2.2 | | ı | |
| 20. Does the Laboratory add 5 ml of SnCl ₂ solution to all standards and samples just before each is to be analyzed? | Sec 11.2.3 | | | |
| 21. Does the Laboratory analyze an Instrument Performance Check (IPC) solution immediate following each calibration, after every 10 th sample, and does it pass (±5% initial, ±10% for all others)? | Sec 9.3.4 | | | , |

| General Comments: | |
|-------------------|--|
| | |
| | |
| | |

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Appendix B: General Walkthrough Checklist

Georgia Department of Natural Resources

Environmental Protection Division Laboratory

Supporting Data and System Audit Walkthrough Checklist

| Laboratory Name: | | | | |
|--|---------|---------|------------------|------------------------------|
| Date of Audit: | | | | |
| Auditor(s): | | | | |
| Method: | | | ☐ Check box | if EPA checklist unavailable |
| SOP# & Revision: | | | | |
| Batch: | | | | |
| QA Sample: | _ | | | |
| Supervisor: | | | | |
| Primary Analyst: | | | | |
| IDCs for Supervisor completed? | Voc | No | NA E | ffective date: |
| IDCs for Primary Analyst completed? | | | | ffective date: |
| CDCs for Supervisor current? | | | | ffective date: |
| CDCs for Primary Analyst current? | | | | ffective date: |
| MDLs for Primary Instrument current? | | No_ | | ffective date: |
| MDLs for Secondary Instrument current? | | No | | ffective date: |
| LDR for Primary Instrument current? | | No_ | | ffective date: |
| LDR for Secondary Instrument current? | _ | No_ | | ffective date: |
| RT Studies for Primary Instrument current? | | No No | | ffective date: |
| RT Studies for Secondary Instrument current? | _ | No_ | | ffective date: |
| Control Charts are up-to-date for this method? | | | | ffective date: |
| control charts are up-to-date for this method? | res | . 110 | INA E | nective date: |
| QC Standards associated with batch (curves, spi | kes, et | tc.) va | lid (not expired | d)? Yes No NA |
| QC Standards properly logged in Standard Log B | look? | Yes_ | No_NA_ | |
| Standard Number:trace | able w | ith Ce | ertificate of An | alysis? Yes No NA |
| (or Reagent) | | | | |
| Batch QA Sample Chain of Custody Available for | revie | w? Y | es No NA_ | - |
| Batch QA Sample Login Entry matches Chain of | Custo | y? Y | es No NA_ | |
| Instrument Maintenance Log Completed for Bat | tch? \ | es_ | No NA | |
| Instrument Run Log Completed for Batch? Yes | No_ | _ NA_ | _ | |
| Batch QA Sample LabWorks Entry matches hard | Lony | data? | Ves No N | JΔ |
| Sateri et volimpie Edutivorità Entry Materies mara | сору | aata. | 105_110_1 | ··_ |
| Were samples received and stored at the correct | ct tem | peratı | ıre? Yes No | NA |
| Was the oven kept at the correct temperature (| if appl | icable |)? Yes No_ | _ NA |
| Was the balance checked on day of use (if appli | cable) | ? Yes | No NA | |
| | | | | |

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| Were non-expired Class 1 weights used to verify the balance? Yes No NA |
|--|
| Were thermometers not expired when used? (if applicable)? YesNoNA |
| Was the pipette checked/calibrated on day of use (if applicable)? YesNoNA |
| Was the refrigerator/freezer/oven checked daily (if applicable)? Yes No NA |
| Was the autoclave checked daily (if applicable)? Yes No NA |
| Was the associated data archived appropriately (if applicable)? Yes No NA |
| Was a quarterly ICV analyzed (if applicable, drinking water only)? Yes No NA |
| Was the associated SOP reviewed annually? Yes_No_NA_ (See S: SOP \rightarrow SOP List (year) \rightarrow select tab \rightarrow see "Annual Review" column. If the audit is before June 30 th and the review has not been done, was the review performed in the previous year?) |
| Are the bottles used traceable with a lot number (if applicable)? YesNoNA Are the preservatives used traceable with a lot number (if applicable)? YesNoNA |
| Are waste bottles properly marked? Yes No NA |
| Is the Hazardous Waste Logbook current? Yes No NA |
| Are reagents and other containers properly labeled? YesNoNA |
| Are red-tagged instruments not in use (if applicable)? Yes No NA |
| Is the final report correct with all relevant information? YesNoNA |
| Comments: |

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Appendix C: Internal Audit Deficiency Report Form

Georgia Department of Natural Resources

Environmental Protection Division Laboratory

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Internal Audit Deficiency Report Form

Year:

Auditor(s):

Section I: Instructions and Definitions

This internal audit will identify apparent deviations for GA EPD Laboratory's Quality Systems requirements. This findings report is divided into several sections:

- 1. The General Walkthrough
- 2. Each Method listed by Laboratory/Department. The methods for the various divisions will typically be grouped together: Drinking Water, Air and Hazardous Waste
- 3. Laboratory-Wide Deficiencies and Recommendations
- 4. There are a few definitions that will help with understanding this report:

Deficiency: An exception found with a procedure that is in deviation of method or Quality Systems requirements. This is a serious exception and must be corrected or addressed promptly.

Recommendation: A potential exception that is not clearly defined in a procedure. It is not technically a violation but could be difficult to defend in an external audit.

Clarification: If there are contradicting requirements in the same or complementary documents. For instance, if a SOP lists r = 0.990 and the LQAP lists $r^2 = 0.990$. This needs clarification to determine if it falls within acceptable limits or simply to reduce confusion.

Comment: A comment is simply that. Comments on any part of the findings.

Section II: General Walkthrough

Section III: Individual Methods

Laboratory:

Method:

Section IV: Laboratory-Wide Deficiencies and Recommendations

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Appendix D: Internal Audit Deficiency Response Form

Georgia Department of Natural Resources

Environmental Protection Division Laboratory

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Internal Audit Deficiency Response Form

Year:

Laboratory:

Deficiency Response(s), include Corrective Actions and Dates of Completion

Method:

Corrective Action and Response:

Uncontrolled Copy